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Institute for Materials Research
National Bureau of Standards
Washington, D. C. 20234

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Quarterly Report
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Prepared for
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Metallurgical Analysis of Wear Particles and Wearing Surfaces

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Metallurgy Division, National Bureau of Standards

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Objective

The objective of the program is to characterize the wear particles and surface degradation produced by wear in bearing and gear life tests (conducted by others) in which the effects of several variables on failure of the wearing surfaces have been examined. The information obtained will be correlated with the results of allied studies conducted by others in an attempt to develop an understanding of the processes producing wear and degradation of metal surfaces in sliding, rubbing, rolling, and/or rotating contact and the effects of lubricants, lubricant additives, bearing materials, etc. on these processes. The characterization of the wear particles and surfaces will aid in the establishment of the interrelationships between wear particle shape, size, size distribution, chemical compositions, metallurgical structure, and surface damage prior to surface failure.

RESULTS

Introduction

Previously obtained results on this project have shown that considerable information can be developed from electron microscope studies of metal wear particles. Data on particle size, shape, composition, degree of oxidation, contamination and deformation can be obtained using either scanning or transmission electron microscopy, electron diffraction, and X-ray emission analysis -- singly and in combination. The wear particles are obtained from the lubricating fluid by the Ferrograph instrument (for a description, see the previous quarterly meeting report of October 19, 1973). The resulting

Ferrogram displays the wear particles collected into strings or groups oriented transversely to the oil flow direction as a result of transverse magnetic fields in the instrument. Nonmagnetic particles are generally not collected as the oil traverses the Ferrograph. Thus the instrument serves to select wear particles (from ferrous systems) and to a large extent ignores other particulates. Deviations in this selection process occur when nonmagnetic particles adhere to magnetic particles in some manner and are thus collected. It is necessary to recognize that particles important in the wear process, for example nonmagnetic abrasives, may not be collected by this technique.

Particles having large magnetic moments are deposited first, near the entrance end of the oil on the Ferrogram. This leads to the development of a size gradient along the Ferrogram, larger particles depositing first. When a variety of material types are present in the particulate collection, the sizing process is disturbed since large particles of a material with low specific magnetic moment will deposit further down the Ferrogram. Specific magnetic moment/unit volume values for three relevant materials are: Fe (171 m Tesla), Fe_3O_4 (48 mT), Fe_3C (99 mT). Thus physical sizing may scatter by factors of 4 or more in a collection of particulates of these materials. As we shall see, examination of particles in strings on the Ferrograms shows a considerable size variation.

A new technique was developed to assist in scanning electron microscope (SEM) evaluation of the Ferrograms. Previously we had taken conventional Ferrogram (glass) slides and over-coated them with vacuum deposited carbon in order to obtain sufficient electrical conductivity. Carbon is an excellent coating material in that no interference is presented for subsequent X-ray emission analysis of the metallic particulates. An alternative technique

was developed that involved stripping the particles from the Ferrogram using a plastic film and subsequently carbon coating that film. The film could then be dissolved in a solvent leaving the particle strings intact on a thin carbon substrate. That method is necessary for transmission electron microscope and diffraction studies.

During this period we have examined several Ferrograms made using glass slides previously coated with thin vacuum deposited carbon layers. The oil sample is passed through the Ferrograph with the precoated slide in place. The particles are deposited on the slide in the usual manner. The carbon under-coating does affect the base-line optical density readings of the Ferrogram and in some cases contributed to oil overflow of the barrier strips along the sides of the Ferrogram slide. However, these problems do not appear serious. After usual washing procedures, the Ferrogram was heated for 30 seconds on a laboratory hot plate to remove residual oils and then examined directly in the SEM. While some particle charging was seen in the SEM, it was found that sufficient electrical conductivity was present to permit detailed examination of the particles. No overcoating procedure was applied to these Ferrograms, and hence all observed surface features and details on the particles are original. This new technique should be of value whenever future SEM examination of a Ferrogram is anticipated. Overcoating with another film (carbon or metal) is still possible should it be necessary for some reason. All the results presented in this report were obtained from uncoated Ferrograms, prepared using carbon-undercoated glass slides.

Two particular oil samples have been examined. One oil sample was obtained through Dr. Dalal at SKF and originated from a service tank system that had lubricated rolling bearing tests. The Ferrogram (F-2117) was prepared at Transonics using carbon precoated slides furnished by us. The

same oil had been used previously to prepare F-1851 that was examined and reported on during the proceeding quarter-year period. Those results should be directly comparable with those reported here. The second oil sample was obtained through Transonics from Pratt and Whitney Corporation. The oil was MIL-L-23699 and had lubricated a single ball tester. The test involved a time-to-failure of 9h 55m, an oil temperature of 300°F, and a ball Hertz stress of 600 kpsi maximum. The bearing materials were not specified. The Ferrogram (F-2119) was prepared using carbon precoated slides from an oil sample taken at 8h time (1h 55m prior to failure).

Ferrogram 2117 Examination

Optical examination of F-2117 at Transonics indicated the presence of cutting wear particles, spherical particles and spalls. The entrance end of the Ferrogram is shown in Fig. 1 at a relatively low magnification (200x) covering an area of about 0.5mm square. The remainder of the Ferrogram slide will not be discussed here but contained relatively few strings of small ($\lesssim 1\mu m$) particles. The previous study of F-1851 from this same oil concentrated on smaller particle strings and those results probably are applicable to the remainder of F-2117 slide. Figure 1 shows that some larger particles do not have sufficient conductivity to the carbon-undercoated glass slide and acquire an electrical charge during a slow beam scan, causing streaks on the photographs. This was rarely noted and caused no significant problem. The labeled areas in Fig. 1 are examined at greater resolution in the following photographs. All the SEM work was conducted at 20kV beam potential, beam currents in the range 50 to 200pA, emissive mode imaging, and with the specimen tilted about 30 degrees toward the detector (located in the upward direction in each photograph). Under these conditions the SEM can resolve details down to 500A°. Thus

we should obtain fine detail on particles 1μm (10,000Å) in size and above. Smaller particles than these can still be detected and some features determined.

The region A in Fig. 1 is shown at higher magnification in Fig. 2. Many flake-like particles are seen, the larger ones being about 10μm in lateral dimensions and of the order of 0.1μm thick. Most of the flakes are bent and corrugated with ragged edges and evident surface structures such as lines or bands. Many small particles, less than 1μm in size are also seen. The larger flakes presumably fractured or spalled away from the bearing material during contact wear and stressing. They could subsequently have been further deformed if recirculated through the bearing contact regions by the lubricating oil and even fractured again to produce some of the smaller particles. The expected EHD film thickness would be one-tenth or less the size of these particles (order of 10μ-inch = 0.3μm)

It is of interest to estimate the numbers of particles collected in these strings. Cursory inspection of Fig. 2a indicates about 10^2 particles in the field, ignoring particles below 0.5μm size. Examination of other subfields of Fig. 1 indicates that to within factors of 3, the total particle count in Fig. 1 can be estimated at about 3×10^3 particles. This number should be kept in mind in connection with the validity of conclusions drawn from individual particle analysis and extended to the macro-system. One clear drawback of the Ferrogram technique for microscopic evaluation is seen in Fig. 2. It involves the tendency for particles to clump and overlay one another in the strings. This prevents accurate particle counts and may bias other observations, in particular X-ray emission analyses.

Another region from Fig. 1 is shown in Fig. 3. A spheroidal particle of diameter 1.7μm is found in one string. One also observes substantial differences in contrast between the particles shown in Fig. 3b, as was noted

in the previous project report. The particles showing lower secondary electron emission (grey images), at the right side of the string and just below "C," are probably more heavily oxidized in accordance with the previous results. Both particles show a porous surface structure that is also suggestive of oxide material. It is recalled that the particles have not been overcoated prior to examination, hence the existing surface structures are preserved.

Another region (D) is shown in Fig. 4a and 4b. Flake-like particles are observed in those strings together with several rod-like particles. Note the electron emission differences between the particles in Fig. 4b. The region E from Fig. 1 is shown in greater detail in Fig. 5. One flake-like particle in this string has considerable surface structure that includes several cracks and smaller, attached particles. A spheroidal particle (2.4μm diameter) is also found. Its size is sufficiently large to permit meaningful X-ray emission analysis in that it would contain nearly all the X-ray emitting volume. Fluorescence effects associated with neighboring particles in such a collection could be substantial and require correction. Analysis of this spheroid showed, however, only an iron peak in the X-ray emission spectra.

An example of the results of an X-ray emission analysis is shown in Fig. 6 taken from region F in Fig. 1. Two particle analyses are shown, one from another spheroid about 2.5μm in diameter and the other from an irregularly shaped 3μm particle. The background analysis was obtained from area c. A stationary electron probe was used for analysis, an electron beam potential of 20kV, beam current of 200pA, and a counting time of about 100 seconds. Both particles are iron with no other indicated elements present. The background contains elemental emissions from oil residue material on the substrate, as reported previously.

The final region (G) presented in detail from Fig. 1 is shown in Fig. 7. A small string of seven particles is present containing a spheroid of 4.5μm diameter. One of the particles has a chip-like appearance and another (c) shows significantly lower image contrast. The X-ray spectra from particles a, b, c and background d are shown in Fig. 7C. The spheroid and particle b are iron, however, particle c shows a weak iron line and a significant Si, K, and Zn content. Since these three elements are associated with oil residue, it appears that particle c may be iron oxide that has absorbed a significant amount of oil.

In summary, the bearing test oil sample contains a wide distribution of particle sizes and types. Many examples of flake-like particles are found (perhaps about 25% of the total) in the size range 1 to 10μm. Some elongated, rod-like particles are found. Four spheroidal particles were found (from an estimated total of 3×10^3). Three of those (size 2.4μm, 2.5μm, and 4.5μm) were analyzed by X-ray emission and found to contain only iron. Contrast differences were found among the particles imaged by secondary electron emission as previously reported for F-1851 from this same oil. They are believed associated in some cases with the iron/iron oxide ratio of the particles. Questions concerning the origin of the spheroids, flake-like particles, and other particles may be resolved by associated examination of the worn surfaces and further studies under more carefully controlled wearing conditions. It does not appear possible to conduct sizing, counting, and shape classifying operations on the particles in the Ferrogram due to problems of clustering and overlap. However, redispersion of the particles on the substrate should be possible and could produce a much more "dilute" array for such analysis.

Ferrogram 2119 Analysis

A low magnification view of the entrance end of this Ferrogram is shown in Fig. 8 covering an area about 0.9 by 2.7mm. Three large particle strings are seen and will be examined in more detail. The remaining areas of the Ferrogram contained isolated particles or small particle ($\lesssim$ 1μm) strings and were not examined closely. The particles are in place on the precoated-carbon Ferrogram slide and have not been overcoated.

Region A in Fig. 8 is shown again in Fig. 9. Several examples of long ribbon-like particles are found in that string together with some flake-like particles. Both types have surface structures and markings that suggest plastic deformation and slip bands. Figure 10 shows the region B in more detail. One curled ribbon particle is seen in this string. Several small particle strings are present in this area but also in the area of region A. Comparison of Figs. 9a with 10a and Figs. 9b with 10b shows the particle sizing capability of the Ferrograph instrument in this example. The larger particles predominate in region A, however, many smaller particles (1 to 4μm) appear present in both regions. Those regions are separated by 0.45mm along the Ferrogram. It seems that the larger magnetic moment particles are deposited earlier on the Ferrogram but that smaller moment particles may deposit either (i) adjacent to larger particles (within their strings) near the entrance end or (ii) further down the Ferrogram in strings of like-sized particles. That "early deposition" behavior may result from relatively large magnetic field gradients within the incomplete, forming particle strings.

The final region (A) studied in Fig. 8 is shown in detail in Fig. 11. The larger flake-like particles are clearly present in this region, as is one large, 8μm diameter spheroid. The spheroid is remarkably smooth and free of surface structures as compared to adjacent particles. X-ray emission

spectra from four of the particles are shown in Fig. 12. Particle c produces the usual lone iron peak, however, the spheroid produces a weak iron peak together with a strong silicon peak. None of the other particles analyzed showed a prominent Si peak. The flake-like particle d contains significant amounts of Cr and Ni, suggesting an alloy steel. The globular particle e also contains Cr and Ni together with a significant amount of copper.

X-ray signal imaging studies were conducted on this string using the prominent Fe K_α and Si K_α lines. Fig. 13a shows the area of Fig. 12 using the Fe K_α signal. The particles are predominantly iron while the silicon is located principally in the background. Certain particles in this field show a reduced iron content. Fig. 14a is a higher magnification view of the particles near the spheroid. The Fe K_α and Si K_α scans in Figs. 14b and 14c, respectively, prove the low iron-high silicon content of this particular spheroid.

In summary, only one spheroid has been found among the particles in F-2119. It is over twice as large as those found in the F-2117 examination and it contains a significant amount of silicon. This may be due to a thicker oxide covering on the spheroid in this case, leading to more substantial oil absorption and oil residue content. However, there is no appearance of a rough or cracked oxide covering on the sphere, and adjacent particles in the string that show a greater roughness do not show any significant silicon image (Fig. 14c). Analysis through the volume of the spheroid may be required to resolve this matter. The ribbon-like particles found in this Ferrogram were not observed to the same extent in F-2117, nor were any alloy steel particles seen there. Correlation of these particle analysis results with the bulk material surfaces and structures is required.

Future Plans

It is of interest to examine the bulk materials from which these particles originated. The phases present and their size and structures, may correlate in a useful way with the particle observations made here. Further effort is intended toward shape classification of the string particles, and toward an increased understanding of the particle history as evidenced by their present microstructure. Unusual particle morphologies will be examined, such as spherical and cutting-wear particle shapes.

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